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IS 3205 (1984): Precipitated barium carbonate, technical  
[CHD 1: Inorganic Chemicals]



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**IS : 3205 - 1984**  
( Reaffirmed 1991 )

*Indian Standard*

**SPECIFICATION FOR  
PRECIPITATED BARIUM CARBONATE,  
TECHNICAL**

*( First Revision )*

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**BUREAU OF INDIAN STANDARDS**  
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
NEW DELHI 110002

*Indian Standard*SPECIFICATION FOR  
PRECIPITATED BARIUM CARBONATE,  
TECHNICAL( *First Revision* )

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# *Indian Standard*

## SPECIFICATION FOR PRECIPITATED BARIUM CARBONATE, TECHNICAL

### *(First Revision)*

#### 0. FOREWORD

**0.1** This Indian Standard ( First Revision ) was adopted by the Indian Standards Institution on 19 March 1984, after the draft finalized by the Inorganic Chemicals ( Miscellaneous ) Sectional Committee had been approved by the Chemical Division Council.

**0.2** This standard was first published in 1965 and is being revised in the light of latest technical developments. An additional grade of the material used in TV glass ceramics is being incorporated in this revision.

**0.3** Barium carbonate is one of the most important barium chemicals, excluding those which are used for pigments. It is used as a raw material in the manufacture of other barium compounds like barium chloride, barium nitrate, barium sulphate, barium hydroxide, barium oxide, etc. Barium carbonate is used in large quantities in conjunction with hydrochloric acid for removal of sulphate ions from sodium chloride ( brine ) used in the electrolytic cells for the manufacture of caustic soda. It is also used in the removal of sulphates ( present as an impurity in small amounts ) from water together with the precipitation of associated heavy metals, alkaline earth metals and magnesium ions as their corresponding carbonates. The other important uses to which it is put are as a flux in the ceramic industry, as an ingredient in the optical glasses and special fine glassware, in the treatment of steel required for the manufacture of high resistance steel articles, like ball bearings, in the manufacture of heat treatment and case hardening compounds and as an ingredient in the composition used for rat poisoning compound.

**0.4** For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960\*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

\*Rules for rounding off numerical values ( revised ).

## **1. SCOPE**

**1.1** This standard prescribes the requirements and the methods of sampling and test for precipitated barium carbonate, technical.

## **2. GRADES**

**2.1** There shall be two grades of the material, namely :

Grade 1 for use in electro-ceramics,

Grade 2 for use in the manufacture of barium salts, removal of sulphations, as a flux in the ceramic industry, glassware, high resistance steel, heat treatment compounds and as rat poisoning compound.

## **3. REQUIREMENTS**

**3.1 Description** — The material shall be in the form of white powder or friable mass.

**3.2** The material shall comply with the requirements prescribed in Table 1 when tested according to methods given in Appendix A. Reference to the relevant clauses of Appendix A is given in col 5 of the table.

## **4. PACKING AND MARKING**

**4.1 Packing** — The material shall be packed in jute bags lined with polyethylene films or HDPE bags. The bags shall be securely closed.

**4.2 Marking** — The bags shall bear legibly and indelibly the following information :

- a) Name and grade of the material;
- b) Name of the manufacturer and his recognized trade-mark, if any;
- c) Gross and net mass;
- d) Date of manufacture;
- e) Batch number; and
- f) Poison.

### **4.3 BIS Certification Marking**

The product may also be marked with Standard Mark.

**4.3.1** The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

## **5. SAMPLING**

**5.1** The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed in Appendix B.



TABLE 1 REQUIREMENTS FOR PRECIPITATED BARIUM CARBONATE, TECHNICAL

( Clause 3.2 )

Sl No.	CHARACTERISTIC	REQUIREMENT		METHOD OF TEST, REF TO CL NO. OF APPENDIX A
		Grade 1	Grade 2	
(1)	(2)	(3)	(4)	(5)
i)	Barium ( as Ba ), percent by mass, <i>Min</i>	68.2	68.2	A-2
ii)	Carbonate ( as CO <sub>3</sub> ), percent by mass, <i>Min</i>	29.8	29.8	A-3
iii)	Moisture, percent by mass, <i>Max</i>	0.20	0.25	A-4
iv)	Matter insoluble in hydrochloric acid, percent by mass, <i>Max</i>	1.0	1.6	A-5
v)	Free alkali	—	To pass test	A-6
vi)	Water soluble sulphide ( as BaS ), percent by mass, <i>Max</i>	0.3	0.4	A-7
vii)	Total sulphur ( as SO <sub>4</sub> ), percent by mass, <i>Max</i>	0.70	—	A-8
viii)	Alkali and other metalsulphates ( as BaSO <sub>4</sub> ), percent by mass, <i>Max</i>	0.01	2.0	A-9
ix)	Sodium oxides ( as Na <sub>2</sub> O ), percent by mass, <i>Max</i>	0.01	—	A-10
x)	Iron ( as Fe <sub>2</sub> O <sub>3</sub> ), percent by mass, <i>Max</i>	0.01	0.04	A-11
xi)	Tap density, g/ml	1.0 to 1.5	—	A-12
xii)	Residue retained on 63 micron IS sieve, percent by mass, <i>Max</i>	0.1	—	A-13
xiii)	Reaction efficiency ( after 45 minutes ), percent by mass, <i>Min</i>	—	85	A-14

## APPENDIX A

(Clause 3.2 and Table 1)

### METHODS OF TEST FOR BARIUM CARBONATE, TECHNICAL

#### A-1. QUALITY OF REAGENTS

**A-1.1** Unless otherwise specified pure chemicals and distilled water ( see IS : 1070-1977\* ) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

#### A-2. DETERMINATION OF BARIUM

**A-2.0 Outline of the Method** — Barium is precipitated as barium chromate which is then dissolved in dilute hydrochloric acid and treated with solid potassium iodide. The liberated iodine is titrated against standard sodium thiosulphate solution.

##### A-2.1 Reagents

**A-2.1.1 Dilute Hydrochloric Acid** — 1:1 ( v/v ).

**A-2.1.2 Concentrated Hydrochloric Acid** — see IS : 265-1976†.

**A-2.1.3 Concentrated Ammonium Hydroxide** — 20 percent ( m/m ).

**A-2.1.4 Ammonium Chloride** — solid.

**A-2.1.5 Hydrogen Sulphide** — gas.

**A-2.1.6 Lead Acetate Paper**

**A-2.1.7 Methyl Orange Indicator** — Dissolve 0.1 g of methyl orange in 100 ml of water.

**A-2.1.8 Glacial Acetic Acid** — see IS : 695-1975‡.

**A-2.1.9 Ammonium Acetate Solution** — 30 percent ( m/v ).

**A-2.1.10 Ammonium Chromate Solution** — 10 percent ( m/v ), neutral to methyl orange.

**A-2.1.11 Ammonium Chromate Solution** — one percent ( m/v ), neutral to methyl orange.

**A-2.1.12 Ammonium Carbonate Solution** — approximately 20 percent ( m/v ).

**A-2.1.13 Silver Nitrate Solution** — approximately 2 percent ( m/v )

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\*Specification for water for general laboratory use ( *second revision* ).

†Specification for hydrochloric acid ( *second revision* ).

‡Specification for acetic acid ( *second revision* ).

**A-2.1.14 Dilute Nitric Acid** — approximately 2 N.

**A-2.1.15 Potassium Iodide** — solid.

**A-2.1.16 Standard Sodium Thiosulphate Solution** — 0.1 N.

**A-2.1.17 Starch Indicator Solution**

**A-2.2 Procedure** — Weigh accurately about 0.25 g of the material and dissolve in about 30 ml of dilute hydrochloric acid, add 100 ml of water, boil and filter. Wash the residue with water. Collect the filtrate and the washings in a 500-ml beaker and make it alkaline with concentrated ammonium hydroxide. Add about one gram of ammonium chloride, pass hydrogen sulphide gas and filter. Pass hydrogen sulphide gas through the filtrate again till no turbidity appears. Boil the filtrate to drive away hydrogen sulphide gas and test for complete removal of the gas by leadacetate paper. Neutralize the solution with dilute hydrochloric acid using methyl orange indicator. Add 5 to 6 drops of glacial acetic acid together with ammonium acetate solution to neutralize any free mineral acid. Heat the solution and add an excess of ammonium chromate solution.

**A-2.2.1** Allow the precipitate of a barium chromate to settle for an hour. Filter through a filter paper and wash with one percent ammonium chromate solution until the filtrate is free of soluble strontium and calcium salts which are tested if a portion of the filtrate produces no cloudiness with ammonium hydroxide and ammonium carbonate. Finally wash the residue free of ammonium chromate with water till a portion of the filtrate gives only slight reddish brown colour with silver nitrate solution.

**A-2.2.2** Pierce the filter paper containing the barium chromate precipitate and wash the precipitate into a beaker with hot dilute nitric acid. Dilute the solution to about 200 ml and boil. Add ammonium acetate solution to neutralize any free nitric acid. Heat and add sufficient ammonium chromate solution. Filter and repeat washings as in **A-2.2.1** above. Dissolve the precipitate of barium chromate finally in 50 to 100 ml of dilute hydrochloric acid, add about 2 g of solid potassium iodide and allow to react for ten minutes. Titrate the liberated iodine with standard sodium thiosulphate, using starch indicator.

### A-2.3 Calculation

$$\text{Barium, percent by mass} = \frac{0.4579 \times V}{M}$$

where

$V$  = volume in ml of standard sodium thiosulphate required for the titration, and

$M$  = mass in g of the material taken for the test.

### A-3. DETERMINATION OF CARBONATES

**A-3.0 Outline of the Method** — Carbonates are determined by absorbing the carbon dioxide evolved in weighed soda asbestos bulbs.

**A-3.1 Apparatus** — The assembly of the apparatus is shown in Fig.1.

#### A-3.2 Reagent

**A-3.2.1 Dilute Hydrochloric Acid** — 1:1 (v/v).

**A-3.2.2 Concentrated Sulphuric Acid** — see IS : 266-1977\*.

**A-3.2.3 Copper Sulphate** — anhydrous.

**A-3.2.4 Anhydrous Magnesium Perchlorate**

**A-3.2.5 Soda Asbestos** — size range 0.7 to 1.2 mm.

**A-3.3 Procedure** — Weigh accurately about 0.5 to 0.75 g of the material and transfer it to the flask and cover with water. Insert the stopper carrying the separating funnel and condenser and connect the latter with *D*, *C* and *B*. Pass air free from carbon dioxide through the system. Close the stopcock in the separating funnel and insert the weighed bulbs *A*, and *A* in the train. Fill half the separating funnel with dilute hydrochloric acid, replace the stopper carrying the air and see that there is free passage for gases through the train. Open the stopcock in the separating funnel and run acid into the flask slowly. When effervescence diminishes start a flow of water through the condenser and heat the flask slowly so as to secure steady but quite ebullition. When all the carbon dioxide has been boiled out of the solution, remove the flame, introduce the current of air and sweep out all carbon dioxide. Disconnect the weighed bulbs, close inlet and outlet tubes and place them in the balance case. When cool, open the stopper momentarily and weigh against a similar tube used as a counterpoise.

#### A-3.4 Calculation

$$\text{Carbonates, percent by mass} = \frac{136.4 \times m}{M}$$

where

*m* = increase in mass in g due to carbon dioxide absorption, and

*M* = mass in g of the material taken for the test.

### A-4. DETERMINATION OF MOISTURE

**A-4.1 Procedure** — Weigh accurately about 5 g of the material in a clean squat type glass weighing bottle, previously dried and weighed. Place the weighing bottle in an oven maintained at  $110^{\circ}\text{C} \pm 2^{\circ}\text{C}$  till constant mass is obtained.

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\*Specification for sulphuric acid ( second revision ).

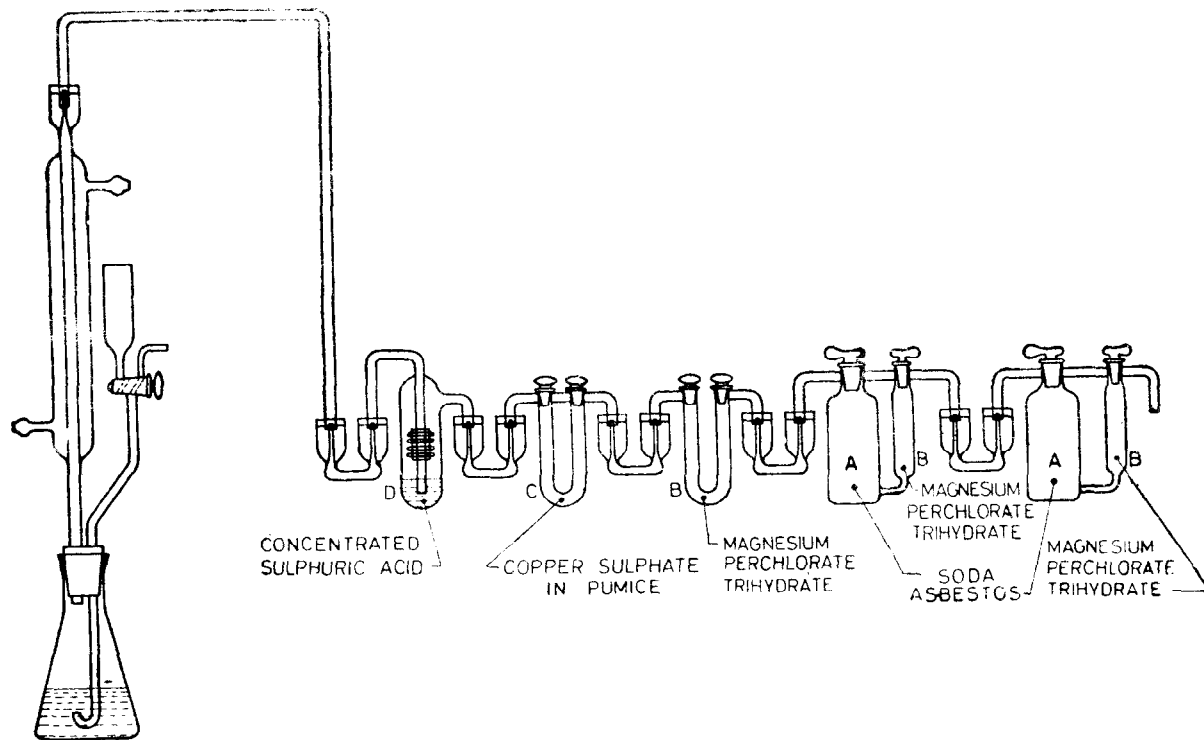


FIG. 1 APPARATUS FOR THE DETERMINATION OF CARBONATES

**A-4.2 Calculation**

$$\text{Moisture, percent by mass} = 100 \frac{(M_1 - M_2)}{(M_1 - M)}$$

where

$M_1$  = mass in g of the weighing bottle with the sample before drying,

$M_2$  = mass in g of the weighing bottle with the sample after drying, and

$M$  = mass in g of the empty weighing bottle

**A-5. DETERMINATION OF MATTER INSOLUBLE IN HYDROCHLORIC ACID****A-5.1 Reagents**

**A-5.1.1 Concentrated Hydrochloric Acid** — see IS : 265-1976\*.

**A-5.2 Procedure** — Mix 10 g of the material with 100 ml of water and add slowly 10 ml of concentrated hydrochloric acid. Dilute with water to 200 ml and heat on the steam-bath for 30 minutes. Filter the hot solution through a previously dried and weighed sintered glass crucible. Wash the residue with water slightly acidified with hydrochloric acid. Again wash the crucible with hot water till the filtrate is free from acid. Place the crucible in an oven maintained at  $110\text{ C} \pm 2^\circ\text{C}$  for two hours. Cool the crucible in a desiccator and weigh.

**A-5.3 Calculation**

$$\text{Matter insoluble in hydrochloric acid, percent by mass} = 100 \frac{(M_2 - M_1)}{M}$$

where

$M_2$  = mass in g of the crucible after filtration,

$M_1$  = mass in g of the crucible before filtration, and

$M$  = mass in g of the material taken for the test.

**A-6. TEST FOR FREE ALKALI**

**A-6.0 Outline of the Method** — Free alkali is determined by titration with standard hydrochloric acid.

**A-6.1 Reagents**

**A-6.1.1 Standard Hydrochloric Acid** — 0.02 N.

**A-6.1.2 Phenolphthalein Indicator** — Dissolve 0.1 g of phenolphthalein in 100 ml of 60 percent rectified spirit.

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\*Specification for hydrochloric acid ( *second revision* ).

**A-6.2 Procedure** — Shake 0.2 g of the material for 5 minutes with 30 ml of carbon dioxide-free water. Filter and add 5 drops of phenolphthalein to 20 ml of the filtrate. Titrate this solution with standard hydrochloric acid.

**A-6.2.1** The material shall be taken to have passed the test, if not more than 0.25 ml of standard hydrochloric acid is required for the titration.

## A-7. DETERMINATION OF WATER SOLUBLE SULPHIDES

**A-7.0 Outline of the Method** — Water soluble sulphides are determined iodimetrically.

### A-7.1 Reagents

**A-7.1.1 Standard Iodine Solution** — 0.01 N ( see IS : 2316-1968\* ).

**A-7.1.2 Concentrated Hydrochloric Acid** — see IS : 265-1976†.

**A-7.1.3 Standard Sodium Thiosulphate Solution** — 0.01 N ( see IS : 2316-1968\* ).

**A-7.1.4 Starch Indicator Solution** — Triturate 5 g of starch and 0.01 g of mercuric iodide with 30 ml of cold water and slowly pour it with stirring into one litre of boiling water. Boil for three minutes. Allow to cool and decant off the supernatant clear liquid.

**A-7.2 Procedure** — Weigh accurately about 5 g of the material, add 100 ml of water, warm slightly and filter. Wash the residue with hot water and cool the filtrate. Transfer the filtrate to a conical flask containing a measured quantity of 50 ml of standard iodine solution, then add 10 ml of concentrated hydrochloric acid and titrate the excess of iodine with standard sodium thiosulphate solution using starch indicator. Carry out the blank determination simultaneously under similar conditions.

### A-7.3 Calculation

$$\text{Water soluble sulphides ( as BaS ),} = \frac{0.0847 \times (V_1 - V_2)}{M_1}$$

percent by mass

where

$V_1$  = volume in ml of standard sodium thiosulphate solution used in the blank titration,

$V_2$  = volume in ml of standard sodium thiosulphate solution used in the titration, and

$M_1$  = mass in g of the material taken for the test.

\*Methods of preparation of standard solution for colorimetric and volumetric analysis ( *first revision* ).

†Specification for hydrochloric acid ( *second revision* ).

## A-8. ESTIMATION OF TOTAL SULPHUR AS SULPHATE ( $\text{SO}_4$ )

### A-8.1 Apparatus

A-8.1.1 *Beaker* — 400 ml capacity.

A-8.1.2 *Platinum Crucible* — 25 ml.

A-8.1.3 *Volumetric Flask* — 250 ml.

### A-8.2 Reagents

A-8.2.1 *Concentrated Nitric Acid* — see IS : 264-1976\*.

A-8.2.2 *Concentrated Hydrochloric Acid* — see IS : 265-1976†.

A-8.2.3 *Bromine (saturated)* — see IS : 2142-1974‡.

A-8.2.4 *Sodium Carbonate* — AR grade.

A-8.2.5 *Potassium Nitrate* — AR grade.

A-8.2.6 *Methyl Orange* — Dissolve 0.1 g of methyl orange in 60 ml of rectified spirit and dilute with water to 100 ml.

A-8.2.7 *Barium Chloride Solution* — 0.25 M.

A-8.2.8 *Dilute Hydrochloric Acid* — 0.1 N.

A-8.2.9 *Concentrated Hydrofluoric Acid* — AR.

A-8.2.10 *Concentrated Sulphuric Acid* — see IS : 266-1976§.

**A-8.3 Procedure**—Weigh exactly 5 g of the sample and transfer the sample to a 400-ml beaker. Make it to a paste with water. Then add 15 ml con. $\text{HNO}_3$ , 5 ml of con. $\text{HCl}$  and 0.5 ml of bromine. Boil and allow to stand the covered beaker on steam bath for 30 minutes. Evaporate to dryness. Add 10 drops of con.  $\text{HCl}$ , and dilute it to 200 ml with water. Heat to the boiling point. Allow the covered beaker to stand overnight. Filter through a close filter paper (Whatman 42). Wash with hot water. Discard the filtrate. Ash the precipitate in the paper in a platinum crucible. Fuse the mixture with 3 g of AR sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) and 0.5 g of AR potassium nitrate ( $\text{KNO}_3$ ).

**A-8.3.1** Leach the melted mass with water. Transfer the contents of the crucible to a 250 ml volumetric flask. Fill up to the mark with water and mix. Filter through dry paper (discard the first 10 ml) into a dry volumetric flask of 200 ml up to the mark. Transfer the contents of the 200 ml volumetric flask quantitatively to a 600 ml beaker. Add few drops of methyl orange. Acidify with 6 N  $\text{HCl}$ . Boil to remove  $\text{CO}_2$ . Neutralize and acidify

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\* Specification for nitric acid (second revision).

† Specification for hydrochloric acid (second revision).

‡ Specification for bromine, technical (first revision).

§ Specification for sulphuric acid (second revision).



with 0.1 N HCl. Heat to boil and add while stirring 5 ml of 0.25 M BaCl<sub>2</sub> solution. Allow for overnight settling. Filter the precipitate in Whatman 42 paper, wash with a little hot water till free from chloride. Dry and ignite the paper in platinum crucible.

**A-8.3.2** To the ash add 1 ml of 40% HF, and one drop of con. H<sub>2</sub>SO<sub>4</sub>. Again ignite and weigh.

#### A-8.4 Calculation

$$\begin{array}{l} \text{Total sulphur (as SO}_4\text{), percent} \\ \text{by mass} \end{array} = \frac{M_1 \times 51.44}{M_2}$$

where

$M_1$  = mass in g of the precipitate, and

$M_2$  = mass in g of the material taken for test.

**A-8.5 Sensitivity** — 1 mg to 0.01 percent SO<sub>4</sub>.

### A-9. DETERMINATION OF ALKALI AND OTHER METALS

**A-9.0 Outline of the Method** — Alkali and other metals are determined gravimetrically as sulphates.

#### A-9.1 Reagents

**A-9.1.1 Dilute Hydrochloric Acid** — approximately 5 N.

**A-9.1.2 Dilute Sulphuric Acid** — approximately 5 N.

**A-9.2 Procedure** — Dissolve 5 g of the material in 100 ml of water and 20 ml of dilute hydrochloric acid. Heat to boiling, add 12 ml to 15 ml of dilute sulphuric acid and allow to stand for two hours. Filter, evaporate the filtrate to dryness ignite gently and weigh the residue.

#### A-9.3 Calculation

$$\begin{array}{l} \text{Alkali and other metals (as SO}_4\text{),} \\ \text{percent by mass} \end{array} = \frac{100 m}{M}$$

where

$m$  = mass in g of the residue, and

$M$  = mass in g of the material taken for the test.

### A-10. ESTIMATION OF SODIUM OXIDE BY FLAME PHOTO-METER

#### A-10.1 Reagents

**A-10.1.1 Sodium Chloride** — AR grade.

#### A-10.2 Apparatus

**A-10.2.1 Volumetric Flask** — 250 ml capacity.

**A-10.2.2 Flame Photometer** — with a filter for sodium.

**A-10.3 Stock Solution of Sodium Chloride** — Dissolve 2.545 g of AR grade sodium chloride in distilled water in 1 000 ml volumetric flask and dilute to the mark. Shake well taking care that the solution does not leak or come in contact with fingers. This solution contains 1 mg of Na per ml of the solution.

**A-10.4 Preparation of Calibration Curve**

To prepare calibrating solution containing 2, 4, 6, 8, 10 ppm of sodium transfer 2, 4, 6, 8, 10 ml each of stock solution in 5 different ( one litre volumetric flask by means of pipette and dilute up to the mark with distilled water ). Pour the calibrating solution into 1 000 ml or 500 ml plastic bottles and label them as 2, 4, 6, 8, 10 ppm of sodium respectively. The label may be covered with a transparent cello tape, so that it does not come out after some time.

Bring the sodium filter in flame photometer in position. The galvanometer spot is adjusted to left hand zero, of the scale of galvanometer when distilled water is atomized. Adjust the spot to 100 or 200 as required by atomizing highest concentration (10 ppm solution ). Take the reading in quick succession of the other calibrating solution. Atomize distilled water in between the readings. It is better to repeat the series of readings to feel safe about it.

After all the readings are over, plot the graph of concentration on *X* axis, and galvanometer reading on *Y* axis. This curve is used for determining the sodium concentration of unknown solution.

**A-10.5 Procedure** — 5 g of  $\text{BaCO}_3$  powder taken in a 400 ml beaker. Make it to a paste. Add drop by drop con. AR HCl, to dissolve the  $\text{BaCO}_3$  powder. When all the carbonate dissolved, just neutralize the solution to pH 7 with ammonia solution. Keep the solution in a water bath so that excess ammonia expels out. Now filter the solution through Whatman 42 filter paper into a 250 ml standard volumetric flask, wash well and make up the solution to 250 ml mark. Now take this solution and atomize it in the flame photometer using the sodium filter and note the galvanometer reading for the unknown solution.

From the curve we have already plotted, find out the concentration of sodium for the corresponding galvanometer reading noted in the galvanometer for the unknown solution.

**A-10.6 Calculation**

$$\text{Sodium (as Na) parts per million} = \frac{\text{Concentration} \times 250}{5\text{g}}$$

$$\text{Sodium oxide (as Na}_2\text{O), parts per million} = \text{Na} \times 1.35$$

## A-11. DETERMINATION OF IRON

**A-11.0 Outline of the Method** — Iron is determined colorimetrically by visual comparison.

### A-11.1 Apparatus

**A-11.1.1 Nessler Cylinders** — 100-ml capacity ( *see* IS : 4161-1967\* ).

### A-11.2 Reagents

**A-11.2.1 Concentrated Hydrochloric Acid** — *see* IS : 265-1976†.

**A-11.2.2 Ammonium Persulphate** — solid.

**A-11.2.3 Butanolic Potassium Thiocyanate Solution** — Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient n-butanol to make up to 100 ml and shake vigorously until the solution is clear.

**A-11.2.4 Standard Iron Solution** — Dissolve 0.702 g of ferrous ammonium sulphate [  $\text{FeSO}_4(\text{NH}_4)_2 \cdot \text{SO}_4 \cdot (6\text{H}_2\text{O})$  ] in water, add 10 ml of concentrated hydrochloric acid and dilute to one litre. One millilitre of this solution is equivalent to 0.1 mg of iron ( as Fe ).

**A-11.3** Weigh accurately about 0.25 g of the material and dissolve in 10 ml of concentrated hydrochloric acid and dilute with 10 ml of water. Add 30 mg of ammonium persulphate, warm and filter into a Nessler cylinder. Cool and add 5 ml of butanolic potassium thiocyanate solution. Add 5 ml of butanolic potassium thiocyanate to another Nessler cylinder containing 10 ml of concentrated hydrochloric acid, 30 mg of ammonium persulphate and 10 ml of water. Run in standard iron solution into the second Nessler cylinder from a burette until after thorough mixing the colours in the two Nessler cylinders are matched.

### A-11.4 Calculation

$$\text{Iron ( as Fe}_2\text{O}_3 \text{ ), percent by mass} = \frac{0.0143 V}{M}$$

where

$V$  = volume in ml of standard iron solution required, and

$M$  = mass in g of the material taken for the test.

## A-12. TAP DENSITY

### A-12.1 Apparatus

**A-12.1.1 Dried Measuring Cylinder** — 100 ml capacity.

\*Specification for Nessler cylinders.

†Specification for hydrochloric acid ( *second revision* ).

**A-12.1.2 Rubber Base Pad**

**A-12.1.3 Dry Funnel**

**A-12.2 Procedure** — Take a sufficient quantity of the dried sample and slip it gently and smoothly through the funnel into the measuring cylinder, up to the 100 ml mark without knocking. Give constant tapping on the rubber base pad, slowly and smoothly, so that no sample is getting out while giving tapping. When a constant volume is obtained note down the volume and transfer the sample and weigh the sample accurately.

**A-12.3 Calculation**

$$\text{Tap density, g/ml} = \frac{M}{V}$$

where

$M$  = mass in g of the material, and

$V$  = volume in ml of the material.

**A-13. RESIDUE ON SIEVE**

**A-13.1 Apparatus**

**A-13.1.1 Sieve** — 63 micron IS sieve.

**A-13.2 Reagents**

**A-13.2.1 Refined Linseed Oil** — See IS : 76 - 1950\*.

**A-13.2.2 Petroleum Hydrocarbon Solvent** — 145/205.

**A-13.3 Procedure** — Weigh accurately the appropriate quantity of the sample dried at  $110 \pm 2^\circ\text{C}$  for 2 hours. Transfer the sample to a glazed porcelain plate. Thoroughly incorporate a sufficient quantity of refined linseed oil with a palette. Knife without any grinding action until a uniform paste is obtained. Mix the paste with 100 ml of petroleum hydrocarbon solvent 145/205 and transfer the mixture on to a 63 micron IS sieve by rinsing with further quantities of petroleum hydrocarbon solvent. Wash the residue left on the sieve with petroleum hydrocarbon solvent gently brushing with a soft camel hair brush until the washings are clear. Dry the residue to a constant mass at  $100 \pm 2^\circ\text{C}$ .

**A-13.3.1** Express the result at the percentage of the mass of the material taken for the test.

**A-14. REACTION EFFICIENCY**

**A-14.1 Reagents**

**A-14.1.1 Dilute Sodium Sulphate Solution** — 2 percent.

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\*Specification for linseed oil, refined, for paints.

**A-14.1.2 Standard Hydrochloric Acid — 1 N.****A-14.1.3 Methyl Orange Indicator**

**A-14.2 Procedure** — Take 1 g of the sample and add to it dilute sodium sulphate solution in a 250 ml beaker. Boil for 45 minutes. Cool and filter. Titrate the filtrate against standard hydrochloric acid using methyl orange as indicator, the following reaction takes place:

**A-14.3 Calculation**

Reaction efficiency, percent by mass =  $V \times 987$

where

$V$  = volume in ml of standard hydrochloric acid required.

## APPENDIX B

(Clause 5.1)

### SAMPLING OF PRECIPITATED BARIUM CARBONATE, TECHNICAL

**B-1. GENERAL REQUIREMENTS OF SAMPLING**

**B-1.0** In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

**B-1.1** Samples shall be drawn in a protected place not exposed to damp air, dust or soot.

**B-1.2** The sampling instrument shall be clean and dry when used.

**B-1.3** Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples, from adventitious contamination.

**B-1.4** To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

**B-1.5** The samples shall be placed in clean, dry and air-tight glass or other suitable containers on which the material has no action.

**B-1.6** The samples containers shall be of such a size that they are almost completely filled by the sample.

**B-1.7** Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

**B-1.8** Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

## **B-2. SCALE OF SAMPLING**

**B-2.1 Lot** — All the packages in a single consignment of the material and from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of packages from different batches of manufacture, the batches shall be marked separately and the packages in each such batch shall constitute separate lots.

**B-2.2** The number of packages to be selected from the lot shall depend upon the size of the lot and shall be in accordance with Table 2.

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**TABLE 2 NUMBER OF PACKAGES TO BE SELECTED**

LOT SIZE	NUMBER OF PACKAGES TO BE SELECTED
$N$	$n$
(1)	(2)
Up to 100	5
101 to 300	6
301 to 500	7
501 to 800	8
801 to 1300	9
1301 and above	10

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**B-2.3** These packages shall be selected at random from the lot and in order to ensure the randomness of selection random number tables may be used. In case random number tables are not available, the following procedure may be adopted.

‘Starting from any package in the lot count them as 1,2,3 . . . up to  $r$  and so on in one order, where  $r$  is integral part of  $N/n$  ( $N$  being the lot size and  $n$  being the number of packages to be selected). Every  $r$ th package thus counted shall be withdrawn to give sample for test.

## **B-3. TEST SAMPLES AND REFEREE SAMPLES**

**B-3.1** Draw with an appropriate sampling instrument small portions of the material from different parts of the packages in the sample. The total quantity of material so drawn from each package shall be approximately three times the quantity required for carrying out the tests for all the requirements.

**B-3.2** Mix thoroughly the portions drawn from the same package to form an individual sample representative of the package. Also mix together equal portions from the individual samples to form a composite sample representative of the whole lot.

**B-3.3** Divide the individual samples and the composite sample into three sets of test samples, each set shall have individual samples representing each package and a composite sample. Transfer these test samples immediately to thoroughly dried sample containers which shall then be sealed air-tight and labelled with all the particulars of sampling given under **B-1.7**. One of these sets shall be for the purchaser, another for the supplier and the third for the referee. Referee samples shall consist of a set of individual samples and a composite sample marked for the purpose. Referee samples shall bear the seals of the purchaser and the supplier, and shall be kept at a place agreed to between the two. It shall be used in case of dispute between the two.

#### **B-4. NUMBER OF TESTS**

**B-4.1** Tests for the material insoluble in hydrochloric acid, water soluble sulphides and barium shall be conducted on each of the individual samples in the set of test samples.

**B-4.2** Tests for the remaining characteristics shall be conducted on the composite samples.

#### **B-5. CRITERIA FOR CONFORMITY**

**B-5.1** A lot shall be declared as conforming to the requirements of this specification, if the requirements given in **B-5.2** and **B-5.3** are satisfied.

**B-5.2** The individual samples, when tested for material insoluble in hydrochloric acid, water soluble sulphides and barium shall all satisfy the requirements of the specification individually.

**B-5.3** For the remaining characteristics which have been tested on the composite sample, all the test results shall satisfy the corresponding requirements of the specification.

( Continued from page 2 )

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